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64 Magnetic disk substrate.

(5) A magnetic disk substrate manufactured from ceramics consisting essentially of alumina and/or zirconia by hot press or hot isostatic press. It has a porosity of not more than 0.1%, a thermal expansion coefficient of 70×10⁻⁷/°C - 110×10⁻⁷/°C between room temperature and about 400°C, and Vickers' hardness of not less than 1200. It may contain sintering aids such as MgO, ZrO₂, Y₂O₃, Cr₂O₃, MnO₃, SiO₃, NiO, A1N and TiO₃, with or without composite components such as carbides, nitrides and borides of elements selected from groups Va, Va and VIa of the Periodic Table. A magnetic disk manufactured from this disk substrate has such high surface precision and CSS durability that it may be used as a high recording density magnetic disk of either longitudinal magnetic recording or perpendicular magnetic recording.

AGE A

(1) Field of the Invention

The present invention relates to a disk substrate for a high-recording density magnetic disk having a continuous, thin film of a magnetic medium. More specifically, it relates to a ceramic disk substrate having such high surface precision and hardness that a magnetic disk manufactured therefrom can have extremely high recording density, and that it can be operated at a small flying height of a magnetic head.

(2) Description of the Prior Art

Magnetic disks have recently had increasingly high recording capacity and density. In order to have higher recording density, a magnetic medium layer has become thinner, and the spacing between the magnetic disk surface and a magnetic head has been decreasing.

A magnetic disk is repeatedly brought into contact with a magnetic head during the operation.

- More specifically, the magnetic disk is in contact with the magnetic head while it is not rotating, and as it rotates faster, the magnetic head begins to fly due to its lower surface configuration. The magnetic head is flying over the magnetic disk at a certain distance
- usually called "flying height" or "spacing," while the magnetic disk is rotating at a constant velocity. The head again comes into contact with the magnetic disk when the disk stops. This process is called a contact-

start-stop (CSS) cycle. To withstand the impact caused by contact with a magnetic head during the CSS cycles, the magnetic disk substrate is required to have sufficiently high hardness.

Moreover, since the magnetic disk is rotated under the conditions of a very small flying height of a magnetic head, a good surface precision such as small surface roughness and undulation is also required for the magnetic disk. In addition, large pores on the surface cause recodring errors if a pore size is almost as large as one tenth of a bit size. The surface precision is thus an important factor for increasing a magnetic recording density of the magnetic disk. Furthermore, when a thin magnetic film is formed on the 15 disk substrate, for instance, by a sputtering method to provide a so-called thin-film magnetic disk, the magnetic disk is usually subject to heat treatment at temperatures up to about 400°C. So the disk substrate should have a thermal expansion coefficient which is sufficiently close to that of a magnetic medium layer formed thereon.

One target of a high recording density for a magnetic disk for longitudinal (in-plane) magnetic recording (hereinafter referred to simply as

25 "longitudinal magnetic recording disk") is as high as more than 3 x 10³ bits/mm². For this target, the thickness of a thin magnetic film formed on a disk substrate should be less than 0.2 µm and the spacing

should be less than 0.2 μm. The target of a high recording density is much higher for a magnetic disk for perpendicular magnetic recording (hereinafter referred to simply as perpendicular magnetic recording disk"), more than 7,75 x 10⁴ bits/mm². For this target, the magnetic film thickness and the spacing should be less than 0.2 μm and not more than 0.15 μm, respectively. To achieve these targets, a finished disk substrate, which is to be coated with a thin magnetic film, is required to have at least average roughness Ra of not more than 0.01 μm and short-range undulation of not more than 0.06 μm/4mm. The maximum surface roughness Rmax is desirably less than about 0.1 μm, particularly less than about 0.1 μm,

Recently, a high-purity alloy of Al and

4wt.%Mg of a JIS-5086 series is used for a highrecording density disk substrate. After grinding the
surface of the Al-alloy substrate, an alumite layer is
formed by anodization to provide a required surface

20 hardness. Important factors of disk substrates of this
kind are a thickness of an alumite layer, surface precision, the purity and heat resistance of the alloy,
etc. These factors have close relations to the signal
errors of disk magnetic media, the flyability of a

25 magnetic head, contact-start-stop (CSS) durability,
etc.

An alumite layer has a thermal expansion coefficient which is as low as about a quarter of that

of the aluminum alloy, so the alumite layer tends to have cracks during a heat treatment step in the course of forming a layer of magnetic medium such as Y-Fe₂O₃. Thus, the thickness of the alumite layer affects not 5 only surface hardness of the resulting disk but also surface cracking thereof. to prevent the surface cracking, the alumite layer should be as thin as possible to have increased deformability, because deformation can absorb an internal stress caused by the 10 difference in thermal expansion between the Al substrate and the alumite layer. In fact, the alumite layer is as thin as less than 3 µm to prevent the surface cracking between about 250°C and about 400°C. The alumite layer of such thickness, however, does not give 15 satisfactory surface hardness to the disk substrate. Accordingly, even though a lubricant layer is formed on the magnetic medium surface, the magnetic disk can hardly endure more than 20,000 contact-start-stop (CSS) cycles without deteriorating its magnetic recording 20 properties, especially its output level by 10%.

Ceramics appear to be very promissing as

materials for disk substrates because their sintered

bodies are extremely hard and dimensionally stable.

Particularly, alumina and/or zirconia-base ceramics are

highly suitable for disk substrate in that they have

high density (low porosity) and relatively close ther
mal expansion coefficients to those of magnetic media.

Ceramic disk substrates manufactured simply by sin-

tering under atmospheric pressure, however, do not always have a porosity which can meet the above requirements, but sometimes have as large a porosity as more than 1-2%. Such a large porosity makes it impossible to achieve the required surface precision of average roughness Ra of not more than 0.01 μ m and short-range undulation of not more than 0.06 μ m/4mm, etc.

SUMMARY OF THE INVENTION

An object of the present invention, therefore, is to provide a ceramic magnetic disk substrate
having sufficiently high density, hardness and surface
precision.

Another object of the present invention is to

15 provide an alumina and/or zirconia-base ceramic disk
substrate having high density, hardness and surface
precision, as well as a thermal expansion coefficient
between room temperature and about 400°C which is so
close to that of a magnetic medium film to be formed

20 thereon that the magnetic medium film is never cracked
or broken.

A magnetic disk substrate made of ceramics according to the present invention consists essentially of Al_2O_3 and/or ZrO_2 , having a porosity of not more than 0.1%, a thermal expansion coefficient of 70×10^{-7} /°C - 110×10^{-7} /°C between room temperature and about 400°C, and Vickers' hardness of 1200 or more. Such a magnetic disk substrate can be manufactured

through a hot press or hot isostatic press process.

BRIEF DESCRIPTION OF THE DRAWING

The sole figure is a graph showing a contact-startstop test cycle.

5 DESCRIPTION OF THE PREFERRED EMBODIMENTS

Ceramic materials for the magnetic disk substrate of the present invention consist essentially of alumina, zirconia or alumina and zirconia. To make the magnetic disk substrate harder, alumina and/or zirconia may be combined with one or more composite components, which are carbides, nitrides and borides of elements selected from groups IVa, Va and VIa of the Periodic Table. The preferred composite components are TiC and TiB2. To improve the density of a sintered ceramic body, sintering aids such as MgO, ZrO2, Y2O3, Cr2O3, MnO2, SiO2, TiO2, NiO and AlN may be added alone or in combination in the amount of up to 5 weight% in total. The minimum amount of the sintering aids added may be 0.5 weight%.

20 Ceramics which may be used in the present invention may be classified in four categories:

- 1. Alumina-base ceramics
- Alumina titanium carbide and/or titanium boride-base ceramics
- Zirconia yttria-base ceramics
 - 4. Alumina · zirconia yttria base ceramics

Alumina-base ceramics include up to 5 weight% in total of one or more sintering aids selected from the group

consisting of MgO, 2rO_2 , $Y_2\text{O}_3$, Cr_2O_3 , MnO_2 , SiO_2 , NiO_3 and TiO_2 . The sintering aids are preferably 0.5-5 weight% in total. 2rO_2 is preferably combined with $Y_2\text{O}_3$.

With respect to alumina titanium carbide

5 and/or titanium boride-base ceramics, the total amount of TiC and/or TiB2 is up to 50 weight%, preferably up to 30 weight%. These ceramics may also include up to 5 weight% of one or more sintering aids selected from the group consisting of MgO, ZrO2, Y2O3, Cr2O3, MnO2, SiO2, TiO2, NiO and AlN. These sintering aids are preferably 0.5-2 weight% in total.

Zirconia yttria-base ceramics contain 4-30

weight% Y2O3 based on ZrO2. Y2O3 serves to stabilize

ZrO2, so such sintering aids as mentioned above are not

necessary to increase the density of a sintered body.

However, the sintering aids may be added. Composite

components selected from the group consisting of car
bides, nitrides and borides of elements of Groups IVa,

Va and VIa of the Periodic Table may also be included

in the amount of up to 50 weight%, preferably up to 30

weight% in total. TiC and TiB2 are the desired com
posite components.

With respect to alumina·zirconia·yttria-base ceramics, the alumina content may be up to 95 weight%.

5 Y2O3 is necessary for stabilizing ZrO2. The ratio of Y2O3 to ZrO2 is 4-30 weight%. The Al2O3-ZrO2-Y2O3 ceramics may include one or more composite components selected from the group consisting of carbides, nitri-

des and borides of elements of Groups IVa, Va and VIa of the Periodic Table in the amount of up to 50 weight%, preferably up to 30 weight% in total.

It has been found that a ceramic fine powder mixture of the above-mentioned composition can be formed into a high-density, high-hardness magnetic disk substrate by way of a hot press (HP) method or a hot isostatic press (HIP) method.

In the hot press, the ceramic powder may be
in advance molded into a desired shape. If necessary,
the ceramic powder molding may be placed under reduced
pressure to remove gass entrapped within the molding.
In such an atmosphere, the hot pressing is carried out
at temperatures of 1400°C -1700°C and press pressure of
200 to 400 bar for 1/2-2 hours.

In the hot isostatic press, a ceramic powder is usually molded into a desired shape at room temperature, and then sintered under atmospheric pressure, before undergoing the HIP treatment. The sintered body is isostatically pressed with inert gas such as argon at temperatures of 1200°C - 1600°C and pressure of 1000 - 1500 bar for 1-5 hours.

20

The HIP pross may be preceded by a hot press (HP) process if it is not easy to increase the density of a ceramic sintered body only by a HIP method. The hot press is particularly desirable for Al₂O₃-(TiC and/or TiB₂) ceramics.

Ceramic disk substrates thus prepared are

much superior in hardness and density to those prepared by usual sintering under atmospheric pressure. The ceramic disk substrate thus prepared has an extremely high density of not less than 99.9% of the theoretical value, so a porosity of not more than 0.1%. In addition, it is very hard, having Vickers' hardness (Hv) of 1200 or more.

A thermal expansion coefficient (α) is another important factor of the disk substrate, because the difference in a thermal expansion coefficient between the disk substrate and a magnetic medium layer formed thereon deeply affects how strongly they are adhered to each other. Given a certain difference in α, the adhesion of a magnetic medium layer to a disk substrate generally decreases as the thickness of the magnetic layer increases. The reason therefor is considered that the thicker the magnetic medium layer, the less it can be deformed, which means that it cannot fully absorb an internal stress caused, during heat treatment, due to the difference in α, making it more vulnerable to cracking and failure.

For a longitudinal magnetic recording disk having a thin magnetic medium film whose thickness is usually between 0.03 µm and 0.2 µm, the difference in between the disk substrate and the magnetic medium film should be within ± about 30x10-7/°C. On the other hand, for a perpendicular magnetic recording disk having a ferromagnetic (for instance, Permalloy) under

layer as thick as 0.2 μ m - 0.8 μ m and a magnetic medium (for instance, Co-Cr) upper layer as thick as 0.1 μ m - 0.7 μ m, the difference in α should be smaller, because the total thickness of the two layers is much larger. The desired difference in α is thus within the range of \pm about $20 \times 10^{-7}/^{\circ}$ C.

Various magnetic media and ferromagnetic materials which may be used in the present invention have the following thermal expansion coefficients between room temperature and about 400°C:

Y-Fe₂O₃ 80×10^{-7} /°C Co-Ni 100×10^{-7} /°C - 120×10^{-7} /°C Co-Cr 100×10^{-7} /°C - 120×10^{-7} /°C Permalloy 90×10^{-7} /°C - 110×10^{-7} /°C Cr 70×10^{-7} /°C - 80×10^{-7} /°C

15

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Thus, the ceramic disk substrate should have a thermal expansion coefficient of $70 \times 10^{-7}/^{\circ}\text{C} - 110 \times 10^{-7}/^{\circ}\text{C}$ between room temperature and about 400°C .

Not only the difference in α between a disk substrate and a magnetic layer, but also the thickness of the magnetic layer including a ferromagnetic under layer affect the adhesion therebetween. Specifically, in an alumina-base disk substrate, a 30 to 80 nm sputtered magnetic film of a Co-20wt.%Ni alloy can adhere to the substrate as strongly as 300 bar or more, but when the film thickness increases to 200 to 300 nm, the adhesion therebetween decreases so badly that it is not

always possible to prevent the magnetic film from cracking or peeling off by heat treatment or minor causes such as small foreign matters. In general, when a magnetic film is as thin as 100 nm or less, the adhesion thereof to the ceramic disk substrate of the present invention is very strong even though the difference in α between them is somewhat large within the above mentioned range.

The as-sintered substrate has a very high porosity. For instance, the as-sintered alumina-base ceramic substrate including MgO, AlN, ZrO₂, etc. has a porosity of about 1%. This substrate, if polished, shows the following surface precision: average roughness of 0.02 µm, maximum roughness of 0.2 µm, short-range undulation of 0.07 µm/4mm. It also has hundreds of pores of 3 µm or more per mm².

After HIP treatment, the disk substrate has a decreased porosity. It is to be noted that the porosity affects the level of surface precision. If the porosity is greater than 0.1% the desired level of surface precision could not be achieved.

The disk substrate of the present invention may be produced in the following way.

Ceramic powder materials are prepared and
molded into a desired shape at room temperature. A
ceramic powder molding is placed in an atmosphere of
reduced pressure to remove gass entrapped within the
molding, and then hot-pressed at 1400°C - 1700°C under

the press pressure of 200 to 40C bar for 1/2-2 hours.

Alternatively, the ceramic powder molding may be first sintered at 1400°C - 1700°C under atmospheric pressure and then subjected to hot isostatic press at 1200°C - 1600°C and 1000 to 1500 bar for 1-5 hours. As mentioned above, the HP step and the HIP step may be combined to provide a ceramic disk substrate with an increased density.

10 The substrate thus prepared is subjected to surface treatments: grinding, lapping and polishing. These treatments per se are conventionally known, so explanations thereof will not be made herein. The resulting disk substrate has average surface roughness 15 Ra of not more than 0.01 µm and short-range undulation of not more than 0.06 μ m/4mm. It also preferably has maximum surface roughness Rmax of less than 0.1 µm, particularly less than 0.05 µm. Such a high surface precision, together with a small porosity, is extremely 20 important for a magnetic disk having a high recording density, because the qualities of magnetic recording are highly susceptible to surface irregularities of the disk substrate. Particularly for a perpendicular magnetic recording disk which is expected to have as high recording density as more than 7.75×10^4 bits/mm² and be operated at a spacing of 0.15 um or less, the fact that the disk substrate of the present invention has very high surface precision is highly significant.

For a longitudinal magnetic recording disk, a magnetic medium layer may be formed on the polished surface of the ceramic disk substrate by various methods including sputtering, physical and chemical vapor deposition, plating, coating, etc. In order to provide increased recording density, a sputtering technique is most preferable. The sputtering technique per se is known to those skilled in the art, so no details thereof will be explained herein. A magnetic medium layer formed on the disk substrate by sputtering is generally as thick as 0.03 µm - 0.2 µm.

On the other hand, for a perpendicular magnetic recording disk, a high-permeability under layer is first formed in the thickness of 0.2 μm - 0.8 μm on the disk substrate, for instance, by sputtering. The high-permeability layer may be made of Permalloy. Formed on this layer is a magnetic medium upper layer in the thickness of 0.1 μm - 0.7 μm .

The magnetic layer may be subjected to a bur20 nishing treatment after coating with a lubricant such
as carbon and fluorocarbon polymers. The lubricant
layer is usually as thick as 10 to 60 nm for carbon
and up to a few hundred angstroms for fluorocarbon
polymers.

While the longitudinal magnetic recording disk is rotating, a magnetic head is flying over the disk by 0.15 μm - 0.6 μm . This spacing between the disk and the magnetic head is as small as 0.15 μm or

less when the parpendicular magnetic recording disk is used. It is to be noted that such small spacing cannot be fully realized without using the magnetic disk having the above mentioned surface precision. In fact, the alumite-coated aluminum disk has Ra of 0.03 µm or so and short-range undulation of 0.1 µm/4mm or so, which are too large to meet the spacing requirements of both types of magnetic heads, particularly of the perpendicular type.

The contact-start-stop durability of the magnetic disk according to the present invention is extremely high. It may vary widely depending on what magnetic head is used, but it may generally be said that with respect to CSS durability, the magnetic disk of the present invention is much superior to those conventional magnetic disks made from aluminum substrates. For instance, magnetic disks comprising alumite-coated aluminum substrates show the CSS durability of 10,000 - 20,000 cycles, while the magnetic disk of the present invention shows the CSS durability of 30,000 cycles or more, in case where γ-Fe₂O₃ magnetic medium layers are formed on the disks.

Incidentally, The CSS durability is herein determined as follows: A magnetic disk starts to

25 rotate and its rotational speed increases for 20 seconds to reach the level of 3600 rpm at A as shown in the figure. It rotates on that level for 5 seconds, (A+B), and its speed decreases for 35 seconds to zero (B+C).

It is thereafter stationary for 15 seconds (C→D).

Thus, one cycle of contact-start-stop is completed.

This cycle is repeated until the output of the magnetic disk decreases 10%. The number of such CSS cycles represents the CSS durability.

- Ej -

The present invention will be explained in further detail by means of the following examples.

Example la

Alumina-base ceramic powder materials of
the compositions as shown in Table I were prepared.
Each powder had more the 99.5% purity and not more than
1.0 µm of particle size. Each ceramic powder material
was wet-blended for 24 hours and dried. It was then
granulated and molded at room temperature under the
pressure of 1000 bar to form a disk body of 150 mm

- in outer diameter, 35 mm in inner diameter and 2.3 mm in thickness. The molded disk body was sintered at 1600°C under atmospheric pressure for one hour. The sintered disk body was then subjected to hot isostatic
- press (HIP) at 1500°C and 1000 bar for one hour. It was then lapped and polished mechanochemically to provide a disk substrate with a mirror surface, having an outer diameter of 130 mm, an inner diameter of 40 mm and a thickness of 2 mm.
- 25 With respect to each of the finished disk substrates, porosity was determined by observing a surface thereof by a microscope and by a water substitution method. The porosity is shown in Table I together

with a thermal expansion coefficient (a) and Vickers' hardness (Hv) for each disk substrate.

Table I

		-			
5	No.	Composition (wt.%)	Porosity (%)	(x10 ⁻⁷ °C ⁻¹)	Hv
	1	A1203	0.2	80	1600
	2	Al ₂ O ₃ -0.2%Y ₂ O ₃	0.2	78	1600
	3	A1203-0.6%Y203	<0.1	79	1600
	4	Al ₂ O ₃ -1.0%Y ₂ O ₃	<0.1	80	1600
	5	Al ₂ O ₃ -2%Y ₂ O ₃	<0.1	80	1600
10	6	Al ₂ O ₃ -4%Y ₂ O ₃	<0.1	80	1550
	. 7	Al ₂ O ₃ -7%Y ₂ O ₃	0.3	77	1500
	8	Al ₂ O ₃ -0.1%MgO	0.2	80	1600
	9	Al ₂ O ₃ -0.5%MgO	<0.1	77	1600
7.5	10	Al ₂ O ₃ -1.0%MgO	<0.1	80	1600
15	·11	Al ₂ O ₃ -6%MgO	0.2	. 80	1500
	12	Al ₂ O ₃ -1.0%Cr ₂ O ₃	<0.1	78	1600
	13	Al ₂ O ₃ -1.0%SiO ₂	<0.1	80	1600
	14	Al ₂ O ₃ -1.0%NiO	<0.1	80	1600
20	15	Al ₂ O ₃ -2%MnO ₂	<0.1	81	1500
20	161)	Al ₂ O ₃ -2%TiO ₂	<0.1	80	1400
	172)	Al ₂ O ₃ -2%MnO ₂ -2%TiO ₂	<0.1	80	1250

Note: 1) Sintered at 1500°C, then hotisostatically pressed at 1300°C and 1000 bar.

2) Sintered at 1400°C, and then hotisostatically pressed at 1300°C and
1000 bar.

25

The disk substrate of Sample Nos. 1, 2, 7, 8 and 11 do not satisfy the surface precision requirements of the present invention. It is thus clear that the sintering aids should be 0.5-5 weight% of Al_2O_3 .

5 Example 1b

With respect to the disk substrate of Sample No. 9 in Example la, average roughness Ra, maximum roughness Rmax, short-range undulation (SRU) and defect density (DD) and Young's modulus (E) were measured.

Ra and Rmax were measured by using surface roughness testers (TALYSURF, TALYSTEP and TALYROUND, Taylor Hobson, England), and the defect density was determined by counting the number of pores of more than 3 µm in diameter in a unit area (one mm²). The defect density measurement was conducted at 8 spots located approximately midway between the center and periphary of the disk substrate at 45° radial intervals.

The relationships between the porosity and surface precision were also examined on

25 Al₂O₃-0.5wt.%MgO disk substrates. Comparison was made am ng the following two substrates:

- 1. As-sintered, no HIP
- Full HIP (Sample No. 9 above)

The results are shown in Table II.

Table II

No.	Porosity	Ra	Rmax	SRU	Defect Density
	(\(\frac{1}{8} \)	(µm)	<u>(µm)</u>	(µm/4mm)	(3 µm or more)
1	1	0.02	0.2	0.07	Several hundreds
2	<u><</u> 0.1	0.006	0.04	0.05	<2

The disk substrate (Sample No. 9) was kept at 200°C and was subjected to HF magnetron sputtering with an Fe target in an argon atmosphere containing 50% of oxygen. During the sputtering process, oxidation reaction took place, so an Fe₃O₄ thin film was formed on the substrate surface. The substrate was then heated at 300°C for three hours to oxidize the Fe₃O₄ film to provide a 170 nm thick γ -Fe₂O₃ film. The substrate surface was subjected to a burnishing treatment with a sapphire head of the same type as reported by Nippon Telegraph and Telephone Public Corp., and a 20 nm thick lubricant layer of fluorocarbon polymer (Krytox, du Pont, U.S.A.) was formed on the surface to provide a 20 thin-film magnetic disk of longitudinal magnetic recording. The finished magnetic disk had Ra of 0.011 μm , Rmax of 0.1 μm , and short-range undulation of $0.06 \mu m/4mm$.

A CSS test was performed by the procedure as

25 mentioned above (magnetic disc rotation: 3600 rpm),

using a 3350-type magnetic head (Mn-Zn ferrite core

slider). The test was conducted at various flying

heights. As a result, it was observed that even when

the spacing was as small as 0.15 μm , the magnetic head was flying over the disk stably without contacting with the disk surface. In this case, the CSS durability was as high as 30,000 cycles.

For the purpose of comparison, an aluminum disk having a three-μm alumite surface layer was treated in the same manner as above to provide a thin-film magnetic disk. Though the Al magnetic disk had surface roughness close to that of the magnetic disk of this Example, and showed the stable flying height of 0.2 μm, its CSS durability was about 10,000-20,000.

It is clear from the above comparison that a magnetic disk comprising a high-density (not more than 0.1% porosity), high-hardness Al₂O₃-base ceramic

15 substrate is superior to that of an aluminum substrate in terms of CSS characteristics.

Example 2a

Alumina (titanium carbide and/or boride)-base ceramics were examined.

Ceramic powder materials (purity: more than 99.5%, particle size: not more than 1.0 μm) as shown in table III below were molded into the disk shape as in Exmaple la. Each molding was placed in an atmosphere of reduced pressure to remove the gas entrapped therein, and hot-pressed at 1650°C under the press pressure of 300 bar for one hour. A HIP treatment was then performed at 1500°C and 1000 bar for one hour. The substrate thus prepared was surface-

treated in the same way as in Example 1a. Porosity, thermal expansion coefficient (α) and Vickers' hardness Hv wer measured for each disk substrate. The results are shown in Table III.

	No.	Al ₂ O ₃	oosit TiC	tion(wt.%) Others	Porosity (%)	α (x10 ⁻⁷ /°C)	Hv
	1	94.5	5		0.5MgO	<0.1	78	 1700
					•			
-	2	79.5	20	- :	0.5MgO	<0.1	72	1900
10	3	70	30	-	-	<0.1	70	2000
	4	68.5	30	-	0.5MgO 1.0AlN	<0.1	70	2000
	5	68	30		2SiO ₂	<0.1	70	2000
	6 ⁻	49.5	50	-	0.5MgO	<0.1	70	2100
	7	29.5	70		0.5MgO	0.3	69	2300
15	8	94.5	-	5	0.5MgO	<0.1	79	1700
	9	79.5	-	20	0.5MgO	<0.1	73	1900
	10	69.5	-	30	0.5MgO	<0.1	72	2000
	11	49.5	-	50	0.5MgO	<0.1	72	2100
	12	29.5	· •	70	0.5MgO	0.5	68	2300
20	13	93.5	3	3	0.5MgO	<0.1	77	1700
	14	85.5	7	7	0.5MgO	<0.1	73	1900
	15	75	5	20	- *	<0.1	71	2000
	16	75	20	5	_	<0.1	72	2000
	17	72	14	14	-	<0.1	70	2000
25	18	71	14	14	1Mg0	<0.1	72	2000
	19	68	14	14	4MgO	<0.1	72	2000
	20	71	14	14	14203	<0.1	73	2000
	21	71	14	14	1Cr ₂ O ₃	<0.1	72	2000

	22	71	14	14	15i0 ₂	<0.1	71	1900
	23	71	14	14	lnio	<0.1	72	1950
	24	71	14	14	1MnO2	<0.1	73	1900
	25	71	14	14	lTiO2	<0.1	71	1850
5	26	70	14	14	1zro ₂	<0.1	72	195 <u>0</u>
	27	39.5	20	40	0.5MgO	0.2	70	2100
	28	39.5	40	20	0.5MgO	0.2	70	2100

It was appreciated from the above table that Tic and/or TiB2 should be up to 50 weight% in total.

10 It was further observed that such sintering aids as MgO, AlN, ZrO₂, SiO₂, Y₂O₃, Cr₂O₃, NiO, MnO₂, and TiO₂ were effective for increasing the density of the resulting substrate.

Example 2b

The disk substrate (Sample No. 4) of Example 2a was measured with respect to Young's modulus (E), Ra, Rmax, short-range undulation (SRU) and defect density (DD) (3 µm or more pores).

A magnetic disk was manufactured in the same 25 way as in Example 1b. The resulting magnetic disk had the following characteristics.

Ra 0.005 µm

Rmax 0.05 μm

SRU 0.05 µm/4mm

The CSS test of this magnetic disk conducted in the same way as in Example 1b showed that the stable flying height was 0.13 μm and the CSS durability was 50,000 cycles.

The same CSS test was repeated using a

3370-type thin-film magnetic head (Al₂O₃-30wt.% TiC

slider and Permalloy thin-film). As a result, it was
observed that it had a CSS durability of more than

10 20,000 cycles. On the other hand, an aluminumsubstrate magnetic disk having the same magnetic medium
layer had the CSS durability of as small as 10,000

cycles, when tested with the same 3370-type magnetic
head.

15 Example 2c

The disk substrate (Sample No. 26) of Example 2a was measured with respect to Young's modulus (E), Ra, Rmax, short-range undulation (SRU) and defect density (DD) (3 µm or more pore).

20

200

1.84

· · · · · 5

E $3.9 \times 10^5 \text{ N/mm}^2$

Ra 0.004 µm

Rmax $0.03 \mu m$

SRU 0.04 μm/4mm

DD < one defect/mm²</pre>

A magnetic disk was manufactured in the same way as in Example 1b. The resulting magnetic disk had the following characteristics:

Spacing 0.15 µm

CSS

> 80,000

Example 3a

Alumina·zirconia·yttria-base ceramics were examined.

5 Ceramic powder materials having the compositions as shown in Table IV (purity: more than 99.5%, particle size: not more than 1.0 μm) were molded in the same way as in Example la. Each molding was sintered at various temperatures under atmospheric pressure for one hour, and then subjected to HIP treatments at various temperatures under 1000 bar for one hour. They were surface-treated as in Example la. The finished disk substrates had porosities, thermal expansion coefficients (α) and Hv as shown in Table IV.

15 Table IV

	No.	Compos (wt	. %)		Sintering Temp.	Temp.	Porosity	α	Hv
		A1203	2102	¥203	(°C)	(°C)	(8)	$(x10^{-7}/^{\circ}C)$	
	1	98	1	1	1600	1400	<0.1	. 80	1600
	2	94	5	1	1600	1400	<0.1	81	1600
20	3	88	10	2	1600	1400	<0.1	81	1550
	4	70	30	-	1600	1400	0.3	80	1450
	5	69	30	2	1600	1400	<0.1	82	1500
	6	67	30	3	1600	1400	<0.1	82	1500
	7	· 50	30	20	1600	1400	0.3	82	1500
25	8	45	50	5	1600	1400	<0.1	85	1400
	- 191 -	23	70-	7-	1500	1350	<0.1	. 87	1350
	10	10	83	7 ·	1500	1350	<0.1	90	1300
	11	10	66	14 (10Ti	1450 C)	1300	<0.1	92	1400

0131895

12	10	66	14 (10TiB)	1450	1300	<0.1		92	1400
13	5	87	8	1500	1350	<0.1	2	95	1250
14	, 1 ,	90	9	1500	1350	<0.1		98	1200

It was observed that Y_2O_3 was effective for increasing the density of the Al_2O_3 - ZrO_2 sintered ceramics. The reason therefor is considered that Y_2O_3 stabilizes ZrO_2

Example 3b

The HIP-treated, sintered body of Sample
No. 6 in Example 3a had the following properties:

Young's modulus $3.7 \times 10^{-5} \text{ N/mm}^2$ Ra $0.004 \text{ } \mu\text{m}$ Rmax $0.02 \text{ } \mu\text{m}$

Short-range undulation 0.04 µm/4mm

Defect density* ≤ one defect/mm²

Note*... Measured for pores of not less than

3 µm

A magnetic disk was prepared from this substrate in the same way as in Example 1b. It was observed that the resulting magnetic disk was stably flying at the spacing of 0.13 µm and endured more than 50,000 CSS cycles.

Example 4a

25

Zirconia-base ceramics were examined.

Ceramic powder materials having the compositions as shown in Table V were molded in the same way as in Example la. Each powder material had more than 99.5% purity and not more than 1.0 µm of particle

size. Each of the resulting moldings was then sint red at 1450°C under atmospheric pressure for one hour, and then subjected to hot isostatic press (HIP) at 1300°C and 1000 atms for one hour. The resulting disk substrates were surface-treated as in Example 1a. They had porosities, thermal expansion coefficients (α) and Hν as shown in Table V.

Table V

10	No.	Compositi ZrO ₂	on (wt.%) Y2O3	porosity (%)	α (x10 ⁻⁷ /°C)	Hv
10	1	98	2	0.2	105	1200
	2	93	. 7	<0.1	100	1200
	3	88	12	<0.1	98	1250
	4	82	18	<0.1	97	1250
15	5	65	35	0.5	95	1100

Example 4b

25

The disk substrate of Sample No. 4 of Example 4a had the following properties:

•	E	2.3×10 ⁵ N/mm ²
20	Ra	0.003 μm
	Rmax	0.02 µm
	SRU	0.03 μm/4mm
	DD (not less than 3 µm pores)	<pre>≤one defect/mm²</pre>

A magnetic disk was prepared from this substrate in the same way as in Example 1b. It was observed that the resulting magnetic disk was stably flying at the spacing of 0.13 μ m and endured more than 70,000 CSS cycles.

Example 5

Zirconia-based ceramics containing composite components were examined.

Ceramic powder materials having the com
5 positions as shown in Table VI (purity: more than 99.5%,
particle size: not more than 1.0 µm) were molded in the
same way as in Example 1a. Each molding was placed
under reduced pressure to remove the gas entrapped
therein, and hot-pressed at 1450°C and 300 bar for

10 one hour. The hot-pressed body was then subjected to a
HIP treatment at 1300°C and 1000 bar for one hour.
The resulting disk substrates were surface-treated as
in Example 1a. They had porosities, thermal expansion
coefficients (a) and Vickers' hardness (Hv) as shown in

15 Table VI.

Table VI

	No.	Compo ZrO ₂	sition Y ₂ O ₃	Others	Porosity (%)	α (x10 ⁻⁷ /°C)	Hv
	1	81	. 9	10TiC	<0.1	93	1300
20	2	68	2	30TiC	0.2	87	1350
20	3	65	5	30TiC	<0.1	88	1400
	4	63	7	30TiC	<0.1	87	1400
	5	55	15	30TiC	<0.1	87	1400
	6	30	40	30TiC	0.2	87	1300
25	7 .	63	7.	30TiB ₂	<0.1	88	1450
2,3	8 .	. · 66	14	10TiC+10TiB	<0.1	95	1350

Example 6

45 wt.% of Al₂O₃ powder, 50 wt.% of ZrO₂ powder and 5 wt.% of Y₂O₃ powder were mixed uniformly.

Each powder component had more than 99.5% purity and not more than 1.0 μm of particle size. The ceramic powder mixture was molded in the same way as in Example la, and the resulting ceramic powder molding was sintered at 1600°C under atmospheric pressure for one hour. The sintered ceramic body was then HIP-treated at 1400°C and 1000 bar for one hour. The same working and surface-treatment as in Example la were performed on the HIP-treated, sintered body to provide a disk substrate. It had a porosity of less than 0.1%, Hv of 1400 and a thermal expansion coefficient (α) of 85x10⁻⁷/°C. It also had the following surface precision:

Ra

 $0.003 \mu m$

15

Rmax

0.02 µm

Short-range undulation

 $0.03 \mu m/4mm$

Defect density

(3 μm or more pore) \leq one defect/mm²

RF sputtering was performed with a target of

20 Co and 25 wt.%Ni in an argon atmosphere containing 50%

nitrogen (total pressure 18 m Torr (2,4 Pa)) to form a cobaltnickel magnetic film of 50 nm on the disk substrate.

This was then heat-treated at 350°C in vacuum for three
hours, and a 50 nm carbon protective coating was then

25 formed on the surface by RF sputtering. The disk was
sapphire burnishing head flying by 0.1µm over the disk.

The resulting magnetic disk had the following surface
precision:

Ra

0.006um

Rmax

0.08µm

Short-range

undulation 0.03µm/4mm

A CSS test was performed using a 3350-type Mn-Zp ferrite magnetic head.

It was observed that the magnetic head was flying stably by 0.1µm over the magnetic disk. The CSS test revealed that this magnetic disk could endure 100,000 CSS cycles until its output declined 10%.

Example: 7

100

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10

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A one-um thick Cr under layer was formed on the magnetic disk of Example 6 by RF sputtering, and a 60 rm thick cobalt-nickel magnetic medium layer was formed thereon by sputtering with a target of Co-20wt.% Ni in an argon atmosphere. A 50 rm carbon protective layer was further formed in the disk by sputtering. A burnishing treatment was performed in the same way as in Example 6.

The above procedure was repeated on aluminum disk substrates having a 20µm NiP plating layer and a 10µm alumite layer, respectively and mechanochemically polished.

disks, surface precision, stable flying height and CSS durability were measured. The results are shown in Table VI.

		Table VI			
NO.	Disk substrate	Surface precision of disk substrate (um)	Surface precision of magnetic disk (µm)	Stable flying height (µm)	CSS durability (cycles)
-	Alumina-Zirconia	Ra=0.003 Rmax=0.02 SRU=0.03	Ra=0.008 Rmax=0.05 SRU*=0.06	0.1	110,000
8	NiP-plated aluminum	Ra=0.01 Rmax=0.15 SRU*=0.1	Ra=0.015 Rmax=0.23 SRU*=0.1	0.2	30,000
m	Alumite-coated aluminum	Ra=0.02 Rmax=0.15 SRU*=0.07	Ra=0.03 Rmax=0.25 SRU*=0.12	0.25	30,000
••	Note*	SRU: µm/4mm			0131895

The above comparison clearly shows that a magnetic disk constructed from the alumina-zirconia ceramic substrate according to the present invention is much superior to that of an aluminum substrate in terms of surface precision, flying height and CSS durability.

Example 8

The magnetic disk of Example 6 was used to manufacture a thin-film magnetic head of perpendicular 10 magnetic recording. With a target of 78Ni-14Fe-3Cu-5Mo by weight%, DC magnetron sputtering was conducted to form a 0.5 µm Permalloy layer. A 0.2 µm cobalt-chromium magnetic medium layer was then formed by DC magnetron sputtering with a target of Co-14wt.%Cr.

Thereafter, a 26 nm carbon protective layer was formed thereon. A burnishing treatment was carried out for 5 minutes with a sapphire burnishing head flying 0.05 μm over the magnetic disk. The resulting magnetic disk had the following surface precision:

20

Ra

0.01 µm

Rmax

 $0.06 \mu m$

Short-range

 $0.04 \, \mu \text{m}/4 \text{mm}$

A CSS test was conducted thereon with a 3370-type thin-film magnetic head, which comprised a slider of ZrO2-9wt.%Y2O3 having less than 0.1% porosity (sintered at 1450°C under atmospheric pressure for one hour and HIP-treated at 1300°C and 1000 bar for one hour), and a thin film laminate consisting of a

Permalloy layer, an alumina layer and a Cu coil layer. As a result, the stable flying height was 0.1 μm and the CSS durability was 200,000 cycles.

Incidentally, when a liquid fluorocarbon was coated in the thickness of 10 nm as a lubricant instead of carbon on the Co-Cr magnetic medium layer of the above alumina-zirconia disk, the magnetic disk showed substantially the same CSS durability.

A further CSS test was carried out on the

10 magnetic disk of this Example with various thin-film
magnetic heads whose core sliders were made of CaTiO3,
BaTiO3, Al2O3·TiC, and Al2O3/ZrO2 ceramics, respectively. The CSS durability of this magnetic disk used
with these heads was 30,000-40,000 cycles. It was thus

15 appreciated that the perpendicular recording, thin-film
magnetic head of this Example had extremely higher
resistance to sliding wear when used with a thin-film
magnetic head of zirconia-yttria substrate than with
those of the other ceramic substrates.

As described above, the present invention provides a high-density, high-hardness ceramic disk substrate having a porosity of not more than 0.1%, Vickers' hardness of not less than 1,200, a thermal expansion coefficient of 70x10⁻⁷/°C - 110x10⁻⁷/°C and Young's modulus of more than 2x10⁵ N/mm². A magnetic disk constructed from the disk substrate can be operated stably at a very small flying height of a magnetic head, (0.1 μm or so). Particularly when a

thin film Y-Fe₂O₃ magnetic layer is formed on the disk substrate, the resulting magnetic disk can endure as 2-5 times many CSS cycles as those of conventional disks.

The disk substrate of the present invention has extremely high surface precision, so a magnetic disk manufactured therefrom can have extremely high magnetic recording density.

This makes it possible to provide a small

magnetic disk (for instance, 5% inches or less)

having high recording capacity. A small magnetic disk

is also advantageous in that it does not require so

much costly ceramic materials and can be easily manufactured through hot press or hot isostatic press.

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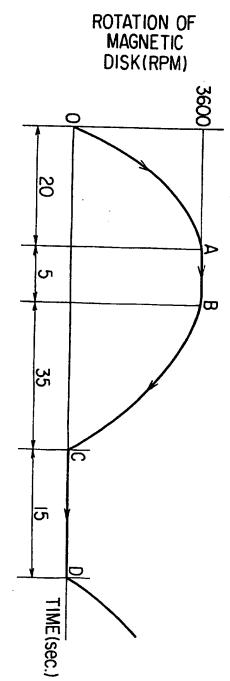
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CLAIMS:

- A magnetic disk substrate manufactured from ceramics consisting essentially of alumina and/or zirconia by hot press or hot isostatic press, having a porosity of not more than 0.1%, a thermal expansion coefficient of 70x10⁻⁷/°C 110x10⁻⁷/°C between room temperature and about 400°C, and Vickers' hardness of not less than 1200.
- 2. A magnetic disk substrate according to claim 1, wherein the alumina-base ceramics comprises up to 5 weights, preferably at least 0.5 weights, in total of one or more sintering aids selected from the group consisting of MgO, ZrO₂, Y₂O₃, Cr₂O₃, MnO₂, SiO₂, NiO, and TiO₂.
 - 3. A magnetic disk substrate according to claim 1, wherein the alumina. (TiC and/or TiB₂)-base ceramics comprises up to 50 weight%, preferably up to 40 weight%, in total of TiC and/or TiB₂ and up to 5 weight%, preferably at least 0.5 weight%, in total of one or more sintering aids selected from the group consisting of MgO, ZrO₂, Y₂O₃, Cr₂O₃, MnO₂, SiO₂, NiO, AlN and TiO₂.
- 4. A magnetic disk substrate according to claim 1, wherein 20 the zirconia.yttria-base ceramics comprises 4-30 weight%, preferably 10-25 weight%, of Y₂O₃.

- 5. A magnetic disk substrate according to claim 4, wherein said zirconia.yttria-base ceramics further comprises up to 50 weight%, preferably up to 30 weight%, in total of one or more composite components selected from the group
 5 consisting of carbides, nitrides and borides of elements of Groups IVa, Va, and VIa of the Periodic Table.
- 6. A magnetic disk substrate according to claim 1, wherein the alumina.zirconia.yttria-base ceramics comprises up to 95 weight% of alumina, and the ratio of yttria to 10 zirconia is 4-30 weight%, preferably 10-25 weight%.
- 7. A magnetic disk substrate according to claim 6, wherein said alumina.zirconia-base ceramics further comprises up to 50 weight% in total of one or more composite components selected from the group consisting of carbides, nitrides and borides of elements of Groups IV, V and VI of the Periodic Table.
- 8. A magnetic disk substrate according to any of claims 1 to 7, wherein said disk substrate has an average roughness of not more than 0.01 μm , and short-range undulation of 20 not more than 0.06 $\mu m/4$ mm.
 - 9. A magnetic disk substrate according to claim 8, wherein said disk substrate has maximum surface roughness of not more than about 0.01 μm_{\star}

10. Use of the magnetic disk substrate according to any of claims 1 to 9 for a longitudinal or a perpendicular magnetic recording disk.





EPO Form 1503 03.82

EUROPEAN SEARCH REPORT

		SIDERED TO BE RELEV	ANT	EP 84108082.3
Category	Cristion of document w of rele	ith indication, where appropriate, want passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CI.4)
A	GB - A - 1 493 GRAPH AND TELEP * Page 1, li	•	1,2,3	G 11 B 5/82
A	GB - A - 1 397 * Claim 1; p	 817 (BASF) age 1, lines 63-6	1-3	
A	<u>US - A - 3 719</u> * Fig. 2 *	525 (PATEL)	1-3	
A	GB - A - 1 257 * Claims 1-1		1	
				TECHNICAL FIELDS SEARCHED (Int. CI 4)
				G 11 B 5/00
			·	
		·		
	The present search report has b	een drawn up for all claims		
	Place of search	Date of completion of the searc		Examiner
	VIENNA	25-10-1984		BERGER
t : part doc A : tech	CATEGORY OF CITED DOCU iccularly relevant if taken alone iccularly relevant if combined w ument of the same category inological background -written disclosure rmediate document	E : earlier ; after th th another D : docume L : docume	e filing date ent cited in the app ent cited for _ther r	ying the invention out published on, or